SeparationTimes

Technically Advanced GC Columns and Supplies for Optimized Performance

Agilent Technologies
Innovating the HP Way

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Our New Look...

Yes, that's right, we've changed and so has the look and feel of our Separation Times newsletter.

What hasn't changed is our commitment to give you practical knowledge about the techniques, methods, theory and products that can make you a better gas chromatographer. The people behind the products haven't changed either. As always, if you don't see what you need here, just give us a call. Allen Vickers and his technical support team still answer the phone everyday with the intention of helping you get the GC results you need and expect.

What has changed as a result of Agilent Technologies and J&W Scientific joining forces is your access to the very best in instrumentation and supplies technology and the world's most popular line of GC columns from a single source. We're now able to provide you with the very best and latest information, knowledge and products designed to provide you with a total solution to virtually every gas phase separation problem that arises.

Finally, I want to make a quick comment about the GC columns offered by Agilent Technologies. Agilent offers the new J&W Scientific brand of GC columns manufactured to the highest specifications in the industry right here in Folsom, California. We have combined the manufacturing of both the traditional lines of DB columns as well as the HP columns into the new J&W brand. The columns are built in the same manner and to the same specifications as always. If you've experienced our columns previously, you will get the same performance and product quality you have come to expect. If you're new to our products, we welcome you with the confidence that you'll be very satisfied with everything we offer. I welcome your comments.

Phil Stremple, Ph.D.
GC Columns Program Manager
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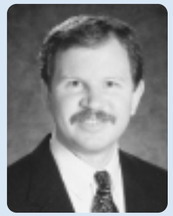


PLOT Columns
for Light Gas Analysis

- **Full family of PLOT phases**
- **Exceptional stability and reproducibility**
- **Expanded offering of column dimensions**

With the joining of Agilent Technologies and J&W Scientific comes a greatly expanded offering of Porous Layer Open Tubular (PLOT) column phases and dimensions for a wide variety of applications. We now offer a comprehensive line of PLOT columns for analysis of fixed gases, low molecular weight hydrocarbon isomers, volatile polar compounds and reactive analytes such as sulfur gases, amines and hydrides. Most of our PLOT phases are offered in dimensions from 0.25 to 0.53 mm ID, allowing for easy column selection for various detector and system requirements. For GC/MS systems, we offer several small diameter columns with truly bonded and immobilized stationary phases, eliminating detector fouling due to particle generation.

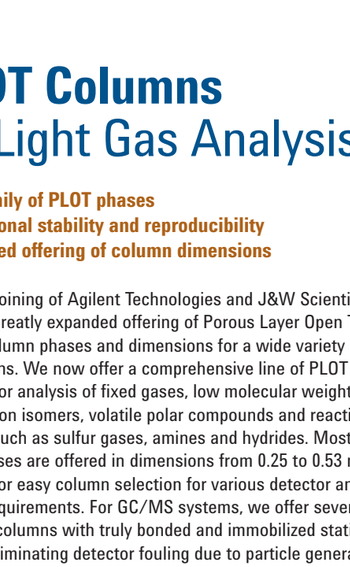
Please review the handy table when selecting a PLOT column for an application.

Ammonia and Water

Column: **HP-PLOT U**
30 m x 0.53 mm I.D.,
20 µm
P/N: **19095-U04**

Carrier: Hydrogen at 7 mL/min
Oven: 150°C isothermal
Injector: Split 25:1, 180°C
Detector: TCD, 200°C
Sample: 2 µL in methanol, 1–10%

1. Air
2. Ammonia
3. Water
4. Methanol



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Column	Stationary Phase	Typical Applications
HP-PLOT Molesieve	5Å molecular sieve zeolite	Permanent and noble gases. Thick and thin films available. Thick film column will resolve argon and oxygen at 35°C.
HP-PLOT Al_2O_3 "KCl"	Aluminum oxide deactivated with KCl	Least "polar" Al_2O_3 phase. Lowest retention of olefins relative to comparable paraffin. C_1 to C_8 hydrocarbon isomers. Column of choice for accurate quantitation of dienes, especially propadiene and butadiene from ethylene and propylene streams.
HP-PLOT Al_2O_3 "S"	Aluminum oxide deactivated with sodium sulfate	Excellent general use Al_2O_3 column. Excellent reproducibility. C_1 to C_8 hydrocarbon isomers. Best for resolving acetylene from butane and propylene from isobutane.
HP-PLOT Al_2O_3 "M"	Aluminum oxide with proprietary deactivation	Most "polar" of the Al_2O_3 columns. Highest retention of olefins relative to comparable paraffin. Excellent general use Al_2O_3 column. Excellent reproducibility. C_1 to C_8 hydrocarbon isomers. Best for resolving cyclopropane from propylene.
GS-Alumina®	Aluminum oxide with proprietary deactivation	Most "polar" of the Al_2O_3 columns. Highest retention of olefins relative to comparable paraffin. Excellent general use Al_2O_3 column. Excellent reproducibility. C_1 to C_8 hydrocarbon isomers. Best for resolving cyclopropane from propylene. Good stability and recovery from water saturation. Selectivity slightly different than "M" column.
HP-PLOT Q	Polystyrene-divinylbenzene	C_1 to C_3 isomers, alkanes to C_{12}, CO_2, methane, air/CO, water, oxygenated compounds, sulfur compounds, solvents.
GS-Q®	Polystyrene-divinylbenzene	C_1 to C_3 isomers, alkanes to C_{10}, CO_2, methane, air/CO. Slightly different selectivity than HP-PLOT Q. Not recommended for quantification of polar compounds.
HP-PLOT U	Divinylbenzene/ethylene glycol dimethacrylate	More polar than HP-PLOT Q and GS-Q. C_1 to C_7 hydrocarbons, CO_2, methane, air/CO, water, oxygenates, amines, solvents, alcohols, ketones, aldehydes.
GS-GasPro	Proprietary, bonded silica-based	C_1 to C_{12} hydrocarbons, CO_2, trace-level sulfurs, hydride gases, inorganic gases, halocarbons, SF_6, oxygen/nitrogen separation at -80°C.
GS-CarbonPLOT	Bonded, monolithic carbon-layer	C_1 to C_5 hydrocarbons, CO_2, air/CO, trace acetylene in ethylene, methane.

ORDER GUIDE*

Phase	I.D. (mm)	Film (µm)	Length (m)	Part Number (P/N)
HP-PLOT Al_2O_3 "S"	0.32	8	50	19091P-S15
HP-PLOT MoleSieve	0.32	25	30	19091P-MS8
HP-PLOT U	0.32	10	30	19091P-U04
GS-GasPro	0.32	N/A	30	113-4332
GS-CarbonPLOT	0.32	1.5	30	113-3132
HP-PLOT Q	0.32	20	30	19091P-Q04

*This is a small sampling of the many PLOT columns and dimensions available. For more information, call Technical Support or go to www.agilent.com/chem

Trademarks
Freon & Vespel — E.I. duPont Nemours & Company, Inc.
Tenax — Enka Research Institute Arnhem
Carbosieve — Supelco
VersaPak — Black & Decker

Total Performance with Superior **CLP Pesticide Columns** DB-35ms and DB-XLB

- **Complete resolution of 22 CLP pesticides in under 16 minutes**
- **Unsurpassed resolution and confirmation of EPA Method 8151A, 8082, 552.2 and 508.1 analytes**
- **360°C upper temperature limit**

By Cameron George
Technical Support Engineer

Accurate identification and confirmation of trace level chlorinated pesticides are difficult tasks facing environmental laboratories. It is imperative that all aspects of the chromatographic system are optimized, including the analytical columns. The GC columns used for these analyses must possess the selectivity, inertness and thermal stability needed to achieve optimum resolution and sensitivity in the shortest time possible. All of these needs are realized with DB-35ms (primary) and DB-XLB (confirmation) columns. The excellent selectivity of high phenyl content phases for chlorinated pesticides is well documented. However, these phases typically suffer from poor thermal stability resulting in high bleed and excessively long analysis times.

DB-35ms utilizes arylene phase technology giving it improved thermal stability through a "stiffening" of the polymer backbone. The result is increased sensitivity and an upper temperature limit of 360°C. Sensitivity is improved through a decrease in the column bleed contribution to background noise, giving a much improved signal-to-noise ratio versus standard 35% phenyl phases. The increased thermal limit translates into shorter analysis times, increased column lifetime and the ability to periodically bake the column at a high temperature to remove semivolatile contaminants.

DB-XLB uses a proprietary second-generation arylene technology giving it the same 360°C upper temperature limit and the lowest bleed of any phase available. Using simple window diagramming, it was possible to identify the exact film thickness necessary to allow DB-XLB to give optimum confirmation power when run using the primary column temperature program.

Figure 1 shows the optimized primary and confirmation chromatograms for the DB-35ms/DB-XLB column pair. Because these GC columns are designed for enhanced thermal fortitude, it is not necessary to bake them excessively upon installation in an attempt to reduce bleed to acceptable levels. A simple 1 to 2 hour conditioning period is typically more than adequate. Conditioning columns overnight, common with cyanopropyl- and trifluoropropyl-containing CLP pesticide columns, is not recommended as it leads to increased column activity and decreased column life.

In short, you are up and running sooner after column installation, with Total Performance intact.

Environmental laboratories are also interested in the analysis of other GC/ECD methods with the same dual column pair used for the chlorinated pesticides. These methods include phenoxy acid herbicides (EPA Method 8151A), haloacetic acids (EPA Method 552.2), PCBs (EPA Method 8082) and EPA Method 508.1 pesticides. This column pair provides baseline resolution of all 8151A analytes on both columns in just over 16 minutes. In addition, DB-35ms and DB-XLB provide the best confirmation and the fewest coelutions of any dual column pair commercially available for 508.1 pesticides. **Chromatograms and analysis conditions for these other common GC/ECD methods can be obtained from Technical Support at 800-552-0413.**

When it comes to environmental analyses, the J&W Scientific brand continues to represent the highest quality GC columns in the world. DB-35ms and DB-XLB are further proof of this as they provide a combination of selectivity, thermal stability and inertness that is unsurpassed in the industry. The result is increased productivity with superior results every time.

ORDER GUIDE*

Phase/Description	I.D. (mm)	Film (µm)	Length (m)	Part Number (P/N)
DB-35ms	0.32	0.25	30	123-3832
DB-XLB	0.32	0.50	30	123-1236
Quartz Deactivated Splitter	–	–	–	705-0903
Deactivated Fused Silica Guard Column	0.53	–	5	160-2535-5

*This is a small sampling of the many DB columns and dimensions available. For more information, call Technical Support or go to www.agilent.com/chem

CLP Pesticides (Figure 1)

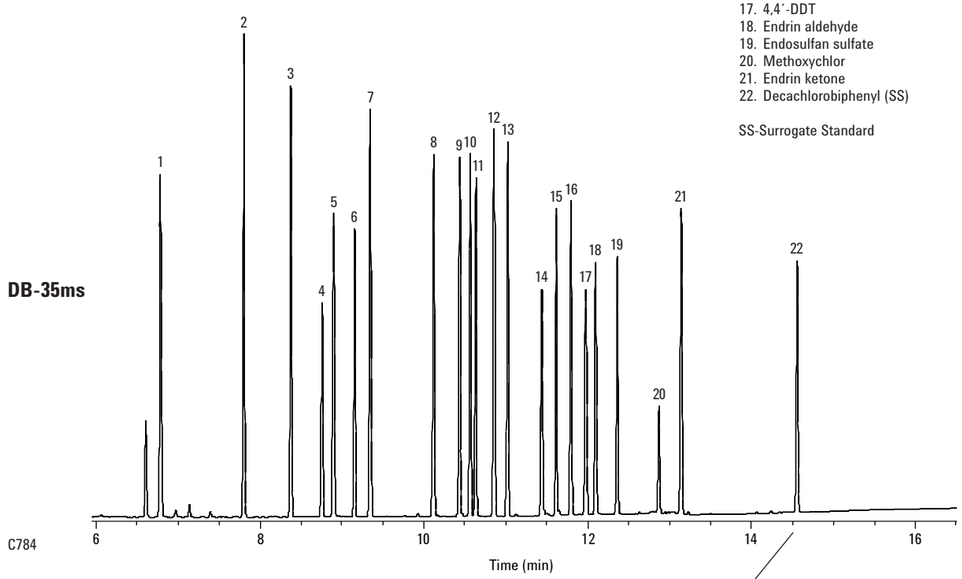
Column: **DB-35ms**
30 m x 0.32 mm I.D., 0.25 µm
P/N: **123-3832**

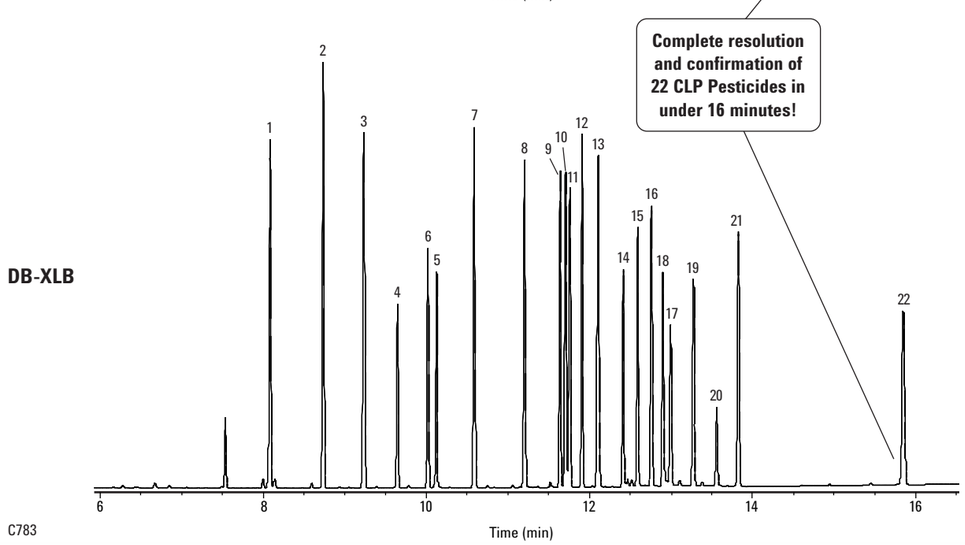
Column: **DB-XLB**
30 m x 0.32 mm I.D., 0.50 µm
P/N: **123-1236**

Carrier: Helium at 45 cm/sec (EPC in constant flow mode)
Oven: 110°C for 0.5 min
110-320°C at 15°/min
320°C for 2 min
Injector: Splitless, 250°C
30 sec purge activation time
50 pg per component
Detector: µECD, 350°C
Nitrogen makeup gas
(column + makeup flow = 30 mL/min constant flow)

1. Tetrachloro-*m*-xylene (SS)
2. α-BHC
3. γ-BHC
4. β-BHC
5. Heptachlor
6. δ-BHC
7. Aldrin
8. Heptachlor epoxide
9. γ-Chlordane
10. α-Chlordane
11. Endosulfan I
12. 4,4′-DDE
13. Dieldrin
14. Endrin
15. 4,4′-DDD
16. Endosulfan II
17. 4,4′-DDT
18. Endrin aldehyde
19. Endosulfan sulfate
20. Methoxychlor
21. Endrin ketone
22. Decachlorobiphenyl (SS)

SS-Surrogate Standard





Air **Analysis** Using DB-1

By Jason Ellis
Technical Support Engineer

- **Huge variety of dimensions for every application**
- **Wide temperature range**
- **Excellent inertness**

DB-1 is considered one of the "workhorses" of the analytical GC world. This extremely stable and durable phase works well for many analyses.

EPA Method TO-14 involves the analysis of volatile air pollutants down to low ppbV/pptV levels using GC/MS and sample preconcentration. *Figure 1* shows the chromatogram of a 400 µL TO-14 air standard, cryogenically concentrated using an Entech Model 7100 and analyzed using a thick film DB-1. A thick film was chosen for this application to provide adequate retention for these volatile analytes. Excellent resolution is obtained for almost all of the 45 compounds including the light gas components (peaks 1-6). While 1,1,2,2-tetrachloroethane (peak 36) and *o*-xylene (peak 37) are not fully resolved by the column these compounds are spectrally resolved using the MSD (m/z 83 and 91, respectively). Resolution between *m*-xylene and *p*-xylene is not required by the method as these compounds are frequently reported together with *o*-xylene as "total xylenes." The use of a DB-1 for this application allows for both TO-14 and ozone precursor analysis without switching columns.

Analysis of sulfur compounds has become a greater issue in recent years with petrochemical composition analysis, industrial hygiene and ambient air odor investigations. *Figure 2* shows a chromatogram of volatile sulfur compounds analyzed using a very thick film DB-1 column and a sulfur selective detector. The high diffusivity of this stationary phase allows for resolution of H_2S and COS on this thick film column with an above ambient starting temperature (50°C). Additionally, excellent peak shape is obtained for active components such as H_2S and the thiols, demonstrating the column's excellent inertness that is required for this demanding analysis.

DB-1 is available in a wide variety of dimensions with over 200 different configurations. With the large selection of dimensions available combined with the tightest QC specifications in the industry, there's a DB-1 for virtually every application.

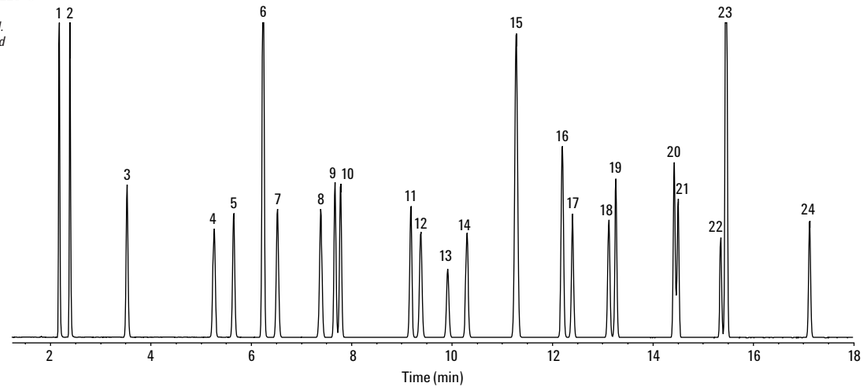
Volatile Sulfur Compounds (Figure 2)

Column: **DB-1**
30 m x 0.32 mm I.D., 5.0 µm
P/N: **123-1035**

Carrier: Helium at 23 cm/sec (H_2S at 50°C)
Oven: 50°C for 4 min, 50-120°C at 20°/min, 120°C for 4 min, 120-220°C at 25°/min, 220°C for 2.5 min
Injector: Split 1:10, 200°C, 600 µL of sulfur gas standard, 3 ppmV each component
Detector: PFPD (OI Analytical), 220°C

1. Hydrogen sulfide
2. Carbonyl sulfide
3. Methyl mercaptan
4. Ethyl mercaptan
5. Dimethyl sulfide
6. Carbon disulfide
7. 2-Propanethiol
8. 2-Methyl-2-propanethiol
9. 1-Propanethiol
10. Ethyl methyl sulfide
11. Thiophene
12. 2-Methyl-1-propanethiol
13. Diethyl sulfide
14. 1-Butanethiol
15. Methyl disulfide
16. 2-Methylthiophene
17. 3-Methylthiophene
18. Tetrahydrothiophene
19. 1-Pentanethiol
20. 2-Ethylthiophene
21. 2,5-Dimethylthiophene
22. 1-Hexanethiol
23. Ethyl disulfide
24. 1-Heptanethiol

Agilent wishes to thank Air Toxics, Ltd. (Folsom, CA) for providing the standard mixture shown in this chromatogram.



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ORDER GUIDE*

Phase	I.D. (mm)	Film (µm)	Length (m)	Part Number (P/N)
DB-1	0.25	0.25	30	122-1032
DB-1	0.25	1.0	30	122-1033
DB-1	0.32	0.25	30	123-1032
DB-1	0.32	5.0	30	123-1035
DB-1	0.32	1.0	60	123-1063
DB-1	0.45	1.27	30	124-1032
DB-1	0.53	3.0	30	125-1034

*DB-1 is available in many more dimensional configurations. For more information, call Technical Support or go to www.agilent.com/chem

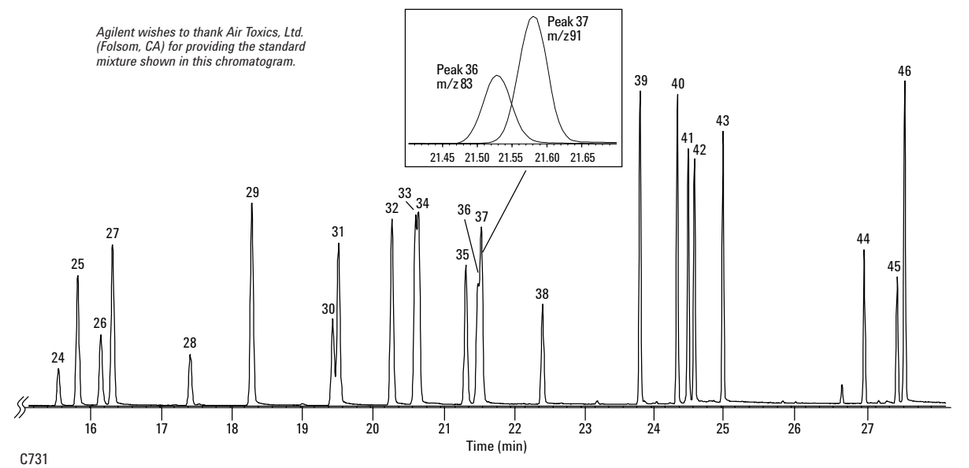
EPA Air Analysis Compendium Method TO-14 Standard (Figure 1)

Column: **DB-1**
60 m x 0.32 mm I.D., 1.0 µm
P/N: **123-1063**

Carrier: Helium at 25 cm/sec measured off of CO_2 at 35°C constant flow mode
Oven: 35°C for 5 min
35-120°C at 5°/min
120-220°C at 30°/min
220°C for 5 min
Injector: Entech 7100 cryogenic sample preconcentrator 400 mL of a 10 ppbV TO-14 standard and 100 mL of a 20 ppbV IS/SS standard
Detector: MSD, full scan of m/z 40-250

1. CO_2
2. Freon 12 (Dichlorodifluoromethane)
3. Chloromethane
4. Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)
5. Vinyl chloride
6. Bromomethane
7. Chloroethane
8. Freon 11 (Trichlorofluoromethane)
9. 1,1-Dichloroethene
10. Methylene chloride
11. Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)
12. 1,1-Dichloroethane
13. *cis*-1,2-Dichloroethene
14. Bromochloromethane (IS)
15. Chloroform
16. 1,2-Dichloroethane
17. 1,1,1-Trichloroethane
18. Benzene
19. Carbon tetrachloride
20. 1,4-Difluorobenzene (IS)
21. 1,2-Dichloropropane
22. Trichloroethene
23. *cis*-1,3-Dichloropropene
24. *trans*-1,3-Dichloropropene
25. 1,1,2-Trichloroethane
26. Toluene-d_8 (SS)
27. Toluene
28. 1,2-Dibromoethane
29. Tetrachloroethene
30. Chlorobenzene-d_5 (SS)
31. Chlorobenzene
32. Ethylbenzene
33. *m*-Xylene
34. *p*-Xylene
35. Styrene
36. 1,1,2,2-Tetrachloroethane
37. *o*-Xylene
38. 4-Bromofluorobenzene (SS)
39. 1,3,5-Trimethylbenzene
40. 1,2,4-Trimethylbenzene
41. 1,3-Dichlorobenzene
42. 1,2-Dichlorobenzene
43. 1,4-Dichlorobenzene
44. 1,2,4-Trichlorobenzene
45. 1,2-Dibromobenzene (IS)
46. Hexachloro-1,3-butadiene

Agilent wishes to thank Air Toxics, Ltd. (Folsom, CA) for providing the standard mixture shown in this chromatogram.



C731

For **Optimum Performance** use Authentic Agilent Replacement Parts and Supplies

The instrument on your bench, much like the one pictured here, represents a significant investment. In fact it costs about as much as a Mercedes SL500. If it was *your* Mercedes SL500, would you use *just any replacement parts* or would you use authentic Mercedes parts? You'd probably use the authentic parts, because you know the same engineers who designed the car, also designed the parts, to deliver optimum performance.

The same thing is true for the instrument on your bench. The Agilent engineers who created this analytical driving machine also specified the parts and supplies required for optimum performance. Each part was designed, tested and manufactured to perform perfectly together. So to keep your instrument running at its very best, consider Agilent specified consumables and replacement parts for the Perfect Fit and optimum performance.

Get it from the Innovators not the Imitators!

Instruments PerfectFit Columns & Supplies

Sample Vials

- Vials, clear wide opening crimp top, write-on spot (100/pk)
  5182-0543
- Crimp caps, silver-aluminum, PTFE/silicone septum (100/pk)
  5182-0552
- 100 µL conical glass microvolume vial inserts, pulled point (100/pk)
  5183-2085

MSD Supplies

- Ion source cleaning kit
  5181-8863
- PFTBA sample, certified (10 g)
  8500-0656
- EI filament
  05972-60053
- Electron multiplier replacement horn
  05971-80103
- Diffusion pump oil (128 mL)
  6040-0819
- Rough pump oil (1 liter)
  6040-0834

Autosampler Syringes

- 5 µL, 23-gauge fixed needle ALS syringe (6/pk)
  5182-0875
- 10 µL, 23-gauge fixed needle ALS syringe (6/pk)
  9301-0725

Gas Purifier

- MegaSorp high capacity carrier gas purifier, 1/8" fittings, removes 0_2, H_20 and hydrocarbons from helium
  RMSH-2
- Split vent kit
  RDT-1020

Inlet Supplies

- Gold plated inlet seal
  18740-20885
- Bleed and Temperature Optimized 11 mm septa (50/pk)
  5183-4757
- High-pressure Merlin Microseal starter kit
  5182-3442
- 4 mm ID straight tube split liner (5/pk)
  210-3003-5
- 4 mm ID single-taper splitless liner (5/pk)
  5183-4695

Column Fittings and Ferrules

- Inlet column hex nut (2/pk)
  5181-8830
- Graphite/Vespel *inlet* ferrules for 250 µm and smaller ID columns (10/pk)
  5181-3323
- Graphite/Vespel *inlet* ferrules for 320 µm ID columns (10/pk)
  5062-3514
- MSD interface column nut
  05988-20066
- Graphite/Vespel *MSD* ferrules for 250 µm and smaller ID columns (10/pk)
  5062-3508
- Graphite/Vespel *MSD* ferrules for 320 µm ID columns (10/pk)
  5062-3506

Water Injection in **Gas Chromatography**

Part 1

By Eberhardt Kuhn
Technical
Support
Engineer

Injection of aqueous samples in gas chromatography is of great interest. In some cases, like purge and trap, injecting water into the column is unavoidable. In other cases, it may be more convenient to inject water directly, rather than going through a cumbersome solvent extraction procedure prior to injection. From a gas chromatographic point of view, water is a less-than-ideal solvent. The problems associated with water include a large vapor expansion volume, poor wetability/solubility in many stationary phases, detector problems and perceived chemical damage to the stationary phase.

The potential problems with water start in the injector. There, samples are vaporized so the analytes can be swept onto the column by the carrier gas. During vaporization in the injector, a sample expands to many times its original liquid volume. Of the commonly used solvents, water has by far the largest vapor expansion volume *(Table 1)*.

Injection liners have volumes ranging from about 200 µL to about 900 µL. That means that the vapor volume of water (assuming a 1 µL injection) exceeds the physical volume of the injection liner. The vapor will then expand outside of the liner *(Figure 1a)*. This phenomenon is called backflash, and can be a problem during subsequent injections. Backflash forces sample components into the gas lines above the liner, such as the incoming carrier gas line and the septum purge line. These gas lines are at much lower temperature, and, thus, the sample will condense *(Figure 1b)*.

From there, the components can re-enter the liner *(Figure 1c)* and show up in subsequent runs (ghost peaks). To minimize backflash problems, use liners with large volumes, make smaller injections, lower the injector temperature, and maintain a high column head pressure and carrier gas flow rate. Or, if possible, use a solvent with a low expansion volume, but this article is about water.

Once the chromatographer has dealt with backflash issues and the sample actually makes it onto the column, water poses still more challenges. Water has a very high surface energy. By contrast, most capillary column stationary phases typically have low surface energies. This results in poor wetting properties of water. Grob[2] and coworkers found, among other results[3], that there were no surfaces that were both wetable by water and sufficiently well deactivated to provide inertness necessary for good chromatography.

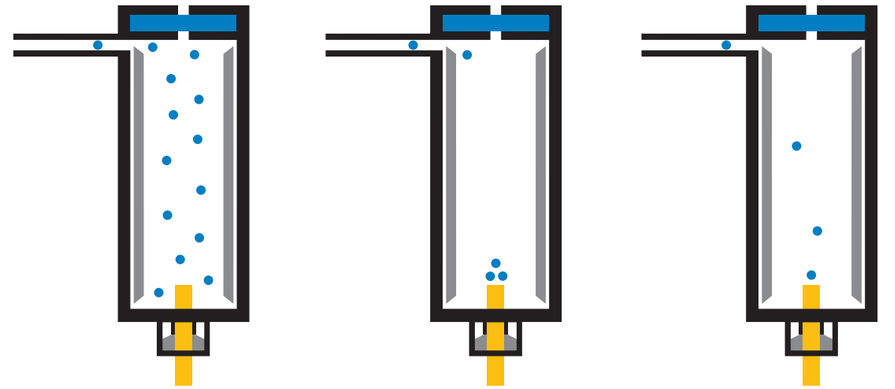
Table 1: Vapor Expansion Volumes of Various Solvents[1]

Solvent	Approx. Vapor Volume (µL)
Isooctane	110
n-Hexane	140
Toluene	170
Ethyl acetate	185
Acetone	245
Methylene chloride	285
Carbon disulfide	300
Acetonitrile	350
Methanol	450
Water	1010

Injection Volume: 1 µL; ***Injector Temperature:*** 250°C; ***Pressure:*** 20 psi

[1] *You can download the Agilent Flow Calculator from our website: http://www.chem.agilent.com/cag/servsup/usersoft/main.html#flowcalc205. This program allows you, among many other things, to calculate the expansion volume of many solvents under your specified conditions.*

Backflash (Figure 1)



a. Vapor expands beyond liner volume

b. Sample condenses in cooler parts of injector

c. Residual sample enters column during subsequent injections

Rather than forming a smooth solvent film that moves uniformly along the column wall, water leaves behind droplets, which can lead to poor and irreproducible chromatography. This behavior is less pronounced on polar stationary phases than on nonpolar ones. As a consequence of this poor wetability, and the high boiling point, part of the water passes through the column as a liquid. Solutes with a high solubility in water may exhibit band broadening and, in severe cases, peak splitting. In the case of on-column injection, nonvolatile compounds, such as salts, can be carried far into the columns by the liquid water plug, increasing the potential for severe column contamination.

In 1981, Schomburg[4] and coworkers published results indicating that water as a solvent is not that bad if the column is properly prepared. Similar results were obtained by Jenkins[5], who also found that the initial oven temperature plays a significant role in performance. Oven temperatures at or above the boiling point of water (100°C) produced better chromatograms (less tailing) than starting at lower temperatures.

Water can also cause problems with detectors. It is not uncommon for water to extinguish an FID flame, especially if the detector gas flows are not set properly. Increasing the hydrogen flow—at the expense of sensitivity—may help stabilize the flame when large water injections are made. Electron capture detectors (ECDs) are also quite sensitive to water vapors. The presence of water lowers the sensitivity of this detector. Jennings[6] and coworkers describe a method to overcome this problem. They used a thick film column to retain their analytes (halogenated hydrocarbons) long enough for the ECD to recover after the water had passed through the detector.

In the second installment in this series about water injections, the issue of stationary phase degradation by water will be discussed.

ORDER GUIDE

Description	Part Number (P/N)
Splitless Liner, single-taper, deactivated, 900 µL volume	5181-3316
Splitless Liner, double-taper, deactivated, 800 µL volume	5181-3315
Glass Union, universal, 2-way (5/pack)	5181-3395
Fused Silica, deactivated 0.53 mm I.D. x 5 m	160-2535-5
Fused Silica, deactivated 0.32 mm I.D. x 5 m	160-2325-5

[2] *Konrad Grob, Jr. and Zhangwan Li, Journal of Chromatography, 473 (1989), pp. 381-422.*

[3] *Although Grob and his group specifically investigated retention gaps (guard columns), their results can be extrapolated to regular columns.*

[4] *E. Bastian, H. Behlau, H. Husman, F. Weeke, and G. Schomburg, in Proceedings of the Fourth International Symposium on Capillary Chromatography, Hindelang, 1981, pp. 465-485, R. E. Kaiser (editor), Hüthig, Heidelberg.*

[5] *R. G. Jenkins, in Proceedings of the Fourth International Symposium on Capillary Chromatography, Hindelang, 1981, pp. 803-815, R. E. Kaiser (editor), Hüthig, Heidelberg.*

[6] *M. Mehran, W.J. Cooper, and W. Jennings, Journal of Chromatographic Science, Vol. 24, (1986), pp. 142-146.*

The **Agilent MSD NoVent**—
A PerfectFit Solution for Saving Time and Money

- **No need to vent MSD for column changes or GC maintenance**
- **Complete column change in just minutes**
- **Extremely inert with no dead volume**

Changing columns in GC/MS systems is a time-consuming process. Venting the MSD, changing the column, pumping down to vacuum and allowing the system to equilibrate can take from several hours to overnight. Instrument downtime means reduced sample throughput and reduced overall productivity.

MSD NoVent is the perfect time-saving solution to this problem. Using a unique purging and restriction design, MSD NoVent allows for the immediate and efficient change of capillary columns with a time savings of more than 95% over the usual method. *Figure 1* shows the change in the nitrogen background signal during a routine column change. It demonstrates how quickly a column can be changed and how quickly the background signal returns to the initial working level. The system is ready to use in minutes.

Figure 2 shows the analysis of common Freons. The lack of peak tailing for these volatile compounds illustrates that MSD NoVent does not introduce any significant dead volume to the chromatographic system.

Maintaining complete system inertness is critical. *Figure 3* shows that MSD NoVent is extremely inert and does not introduce any significant activity to the system. The excellent peak shapes for ppm levels of COS and H_2S are indicative of an inert system. The MSD NoVent transfer line restrictor eliminates head pressure issues, allowing for the use of 0.32 mm I.D. columns shorter than traditionally possible.

In short, Agilent's MSD NoVent provides the solution to a longtime problem with mass spectrometer systems. Column installation and GC maintenance can be performed with more confidence and minimal downtime. The result is increased sample throughput and improved productivity.

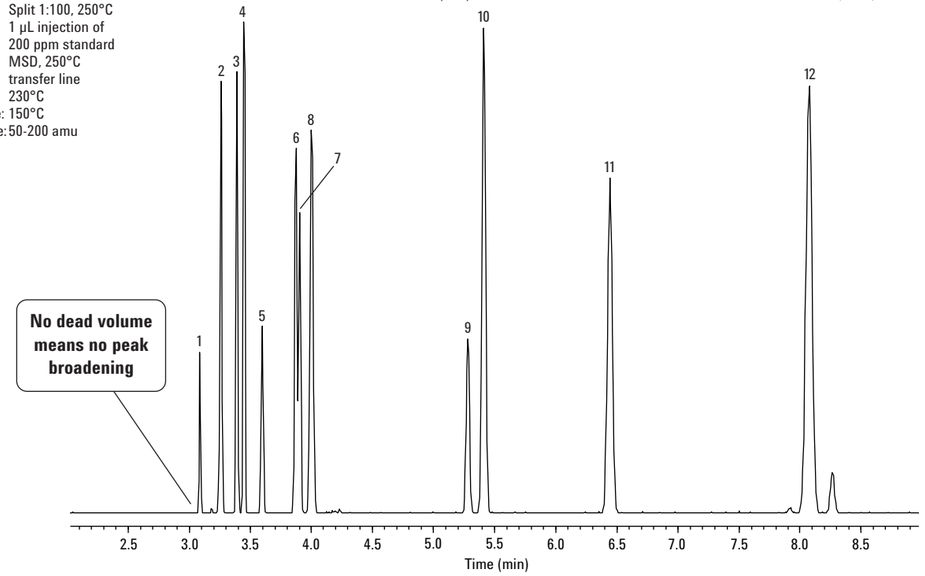
Instruments PerfectFit Columns & Supplies

Freons Using MSD NoVent (Figure 2)

Column: **DB-VRX**
60 m x 0.25 mm I.D., 1.4 µm
P/N: **122-1564**

Carrier: Helium at 35 cm/sec, constant flow mode
Oven: 35°C for 10 min
Injector: Split 1:100, 250°C
Sample: 1 µL injection of 200 ppm standard
Detector: MSD, 250°C transfer line
Source: 230°C
Quadrupole: 150°C
Scan Range: 50-200 amu

1. Trifluoromethane (R23) + Chlorotrifluoromethane (R13)
2. Trifluorobromomethane (R13b1) + 1,1,1,2-Tetrafluoroethane (R134a)
3. 1,1-Difluoroethane (R152a)
4. Chlorodifluoromethane (R22)
5. Dichlorodifluoromethane (R12)
6. 1-Chloro-1,1-difluoroethane (R142b)
7. Chloromethane (R40)
8. 1,2-Dichloro-1,1,2,2-tetrafluoroethane (R114)
9. Chloroethane (R160)
10. Dichlorofluoromethane (R21)
11. Trichlorofluoromethane (R11)
12. 1,1,2-Trichloro-1,2,2-trifluoroethane (R113)



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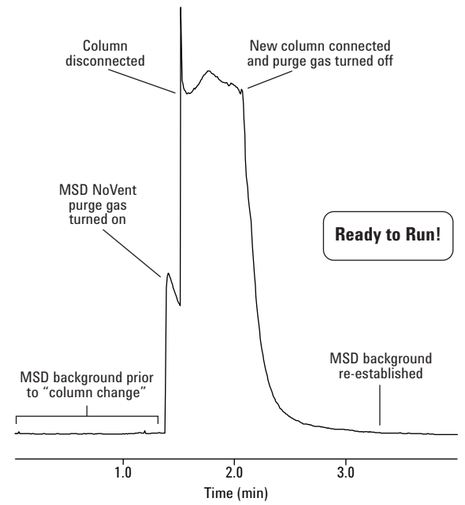
Column Change Scanning for m/z 28* (Figure 1)

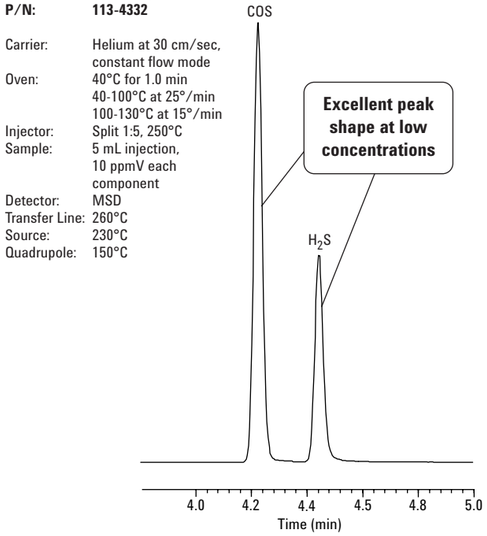


*This procedure was performed with the MSD "on" to demonstrate the MSD NoVent. Do not do this on your MSD as filament damage could result due to improper precautions being taken.

Volatile Sulfur Compounds using MSD NoVent (Figure 3)

Column: **GS-GasPro**
30 m x 0.32 mm I.D.
P/N: **113-4332**

Carrier: Helium at 30 cm/sec, constant flow mode
Oven: 40°C for 1.0 min
40-100°C at 25°/min
100-130°C at 15°/min
Injector: Split 1:5, 250°C
Sample: 5 mL injection, 10 ppmV each component
Detector: MSD
Transfer Line: 260°C
Source: 230°C
Quadrupole: 150°C



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ORDER GUIDE

Description	Part Number (P/N)
6890 MSD NoVent *Includes two each restrictors for 5973 and 5972.*	5183-4782
5890 MSD NoVent *Restrictors not included must be ordered separately.*	5183-4783
Restrictors	
5970	5183-4784
5971/72	5183-4785
5973	5183-4786
GCD	5183-4787

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Chromatographic Ingenuity
DB-VRX: Dreams Made Real

By Allen K. Vickers
Applications
Engineer
Manager

The best thing about being first with a truly "innovative" product is watching others come along afterward with their imitations. Agilent Technologies has certainly brought many truly innovative solutions to the world of gas chromatography that others have tried to copy or "make better." J&W Scientific brand capillary columns are no exception to this history of foreseeing a need and developing a solution that is a marked improvement from previous practice.

In 1985, after the U.S. EPA placed 31 volatile organic compounds (VOCs) on the target list for the GC/MS analysis of EPA Method 624, J&W was quick to introduce DB-624, a capillary column that was far superior in performance to the packed columns recommended in the method.

This was the first commercially available capillary column optimized specifically for the application of EPA Method 624. It was here that polymer chemists sought to design a stationary phase and optimized film thickness to provide the resolving power needed to meet method requirements. Today, DB-624 is still the tried and true choice for the analysis of VOCs in many environmental laboratories.

When the GC/MS instrumentation used for the analysis provided the opportunity to improve sample throughput in the VOC laboratory, once again J&W Scientific came through with a truly original column that sets the high mark for others today. Using computer modeling combined with good old-fashioned chromatographic ingenuity,

DB-VRX gives the fewest chromatographic coelutions and the highest degree of mass spectral integrity for VOC analysis. And while there have been attempts to copy the original, DB-VRX stands out as the clear choice for chromatographers who care about the accuracy and reproducibility of their analytical results.

U.S. EPA Method 8260 Analytes

Column: **DB-VRX**
60 m x 0.25 mm I.D., 1.4 µm
P/N: **122-1564**

Carrier: Helium at 30 cm/sec, measured at 45°C
Oven: 45°C for 10 min
45-190°C at 12°/min
190°C for 2 min
190-225°C at 6°/min
225°C for 1 min
Injector: Purge and trap (O.I.A. 4560)
Sample: 40 ppb per each component in 5 mL water
Purge: Helium for 11 min at 40 mL/min
Trap: Tenax™, Silica gel, Carbosieve™
Desorb: 0.6 min at 220°C
Interface: Split injector at 110°C
with split flow set at 30 mL/min
Detector: MSD, 235°C transfer line
full scan 35–260 amu (m/z 44 subtracted)

ORDER GUIDE*

I.D. (mm)	Length (m)	Film (µm)	Temperature Limits (°C)	Part Number (P/N)
0.25	30	1.40	-20 to 260	122-1534
0.25	60	1.40	-20 to 260	122-1564
0.32	30	1.80	-20 to 260	123-1534
0.45	75	2.55	-20 to 260	124-1574**

*This is a small sampling of the many DB-VRX columns and dimensions available. For more information, call Technical Support or go to www.agilent.com/chem.
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Table of Contents

Page **2** • **Letter from the Editor** *by Phil Stremple*

Pages **2–3** • **PLOT Columns for Light Gas Analysis**

Pages **4–5** • **Superior CLP Pesticide Columns** *by Cameron George*

Pages **6–7** • **Air Analysis** *by Jason Ellis*

Pages **8–9** • **Getting Optimum Performance from Your Instrument**

Pages **10–11** • **Ask Technical Support** *by Eberhardt Kuhn*

Pages **12–13** • **MSD NoVent**

Page **14** • **True Innovators "Dreams Made Real" DB-VRX** *by Allen Vickers*

Cover:

Cover photo was taken by the world renowned chromatographer and action photographer Allen K. Vickers. Extreme biker and Technical Support Engineer, Cameron George, is negotiating some of the technical terrain at Deer Valley Ski Resort (sans snow) in Park City, Utah. Cameron and Allen were in Park City for the 21st International Symposium on Gas Chromatography and Capillary Electrophoresis. No bones were broken, but several rounds of local Wasatch beer met their fate later that evening.

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